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Characterization of cellulose fibers by powder diffraction

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Increasing concerns about the pile-up of plastic garbage on land as well as at sea, have created a growing interest in biodegradable polymer materials. For construction purposes these materials require reinforcement. Here cellulose fibers represent biodegradable alternatives to glass and carbon fibers. This project is focusing on how to extract cellulose fibers from hemp and flax, while retaining the maximum strength of the fibers. The observed strength of cellulose fibers is typically 300 – 1500 MPa, while the theoretical strength is 8000 MPa. Microbial, enzymatic and chemical pre-treatments to remove lignin, pectin and hemicellulose are being tested with respect to effects on strength and interface bonding. An important and traditional method for characterization of cellulose is powder diffraction, which can reveal crystallinity and crystalline domain sizes. However, several problems complicate the evaluation of cellulose powder diffraction patterns: Small crystallite size, typically 4*15 nm, giving very broad and overlapping reflections; preferred orientation due to the fibrous nature of the sample; and transparency effects giving peak shifts and additional asymmetry. As a result a typical powder diffraction pattern will show only a few distinct features (Fig. 1). Several evaluation methods have been used over the years, but since the presentation of the crystal structure of cellulose I β [1], the Rietveld method is the preferred one [2]. Due to the few features in the diffraction patterns the challenge is to minimize the number of refined parameters and thereby correlations between parameters. The transparency peak shift and asymmetry effects have been studied using an internal Si-standard, and crystal domain sizes using pre-oriented samples. We will discuss the effects of sample preparation, peak overlap, transparency effects and preferred orientation, with the overall aim of getting reliable results for crystallinity and crystal domain sizes. The results will be compared to different pre-treatments and fiber strength measurements.

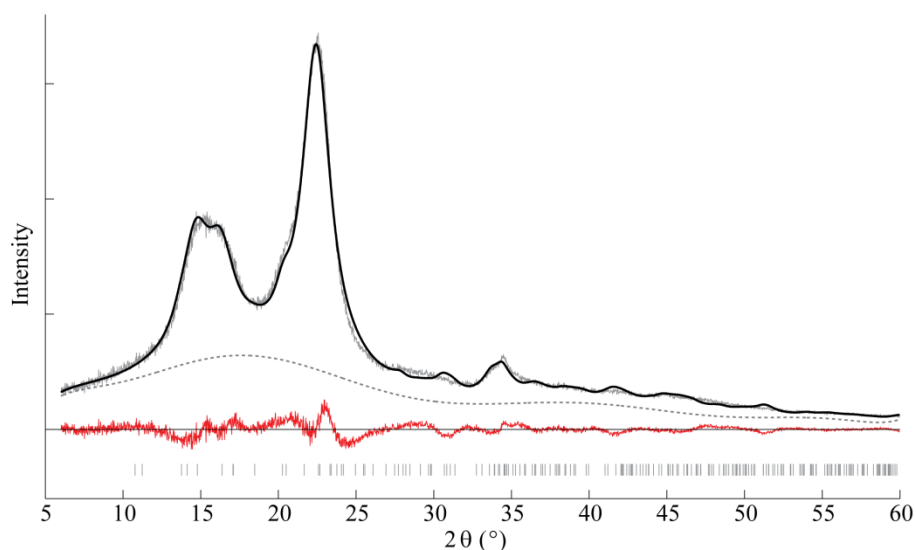


Figure 1. Powder diffraction pattern from raw hemp, 0.5 mm size fraction. Grey: Raw data; black: calculated; red: difference, broken: refined background and Bragg peak markers at the bottom.

[1] Nishiyama, Y., Langan, P. and Chanzy, H. (2002) *J. Am. Chem. Society* 124, 9074 –9082.

[2] Thygesen, A., Oddershede, J., Lilholt, H., Thomsen, A.B. and Ståhl, K. (2005) *Cellulose* 12, 563-576.